

Analysis of Traces in Aqueous Solutions with Thermo Scientific ARL PERFORM'X Advanced WDXRF Spectrometer

Introduction

Analysis of heavy metals and toxic elements as traces in aqueous solutions is becoming increasingly important in view of the environmental concerns. Such analysis demands high sensitivity and accuracy of measurement.

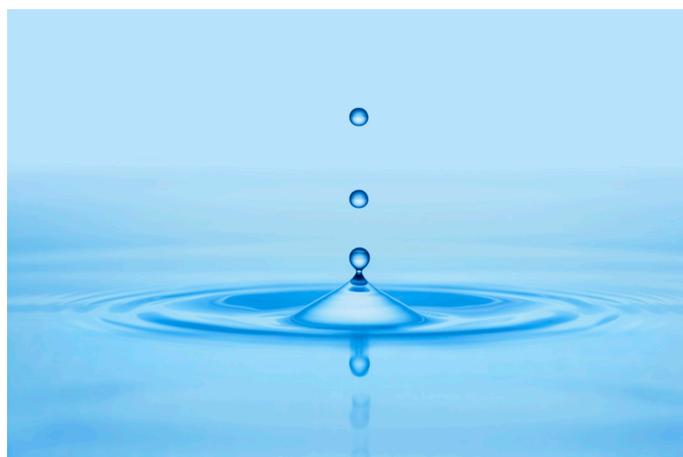
The analysis of liquids can be done with ease and minimum sample preparation in XRF.

Instrument

The Thermo Scientific™ ARL™ PERFORM'X series spectrometer used in this analysis is a 2500 watt system.

This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month. Thanks to a clever management of power, this spectrometer can operate up to 2500W

without requiring external water cooling. The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.



The Secutainer system protects the primary chamber by vacuum collecting any loose powders or liquid in a specially designed container, easily removed and cleaned by the operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. It separates the primary chamber from the goniometer chamber. During liquid analysis helium gas is introduced only in the primary chamber keeping the goniometer under vacuum and a stable temperature. This allows a rapid change-over from vacuum to helium environment, keeps helium consumption to a minimum and ensures the best analytical repeatability. In the "LoadSafe Ultra" optional configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

Sample preparation and results

Solutions with different concentrations of individual elements are prepared by diluting standard solutions with a blank solution. Thus solutions with 2, 5, 10 and 100 ppm were prepared from a standard solution of 1000 ppm and measured in order to derive calibration curves. Limits of detection for the various elements were calculated from these curves.

Figure 1 shows a scan on a sample containing trace amounts of Hg and As in a water solution. Table 1 lists the lines used, crystal-detector combinations and typical limits of detection obtained for the various elements. X-ray tube conditions were set at 60 kV and 41 mA. Cd is measured with a primary beam filter to eliminate overlap from rhodium tube lines.

Conclusion

The analysis of aqueous solutions can be performed rapidly and with ease using the mid power sequential XRF spectrometer ARL PERFORM[®]X at 2500W. Limits of detection for heavy metals and toxic elements range from 0.2 ppm up to 1 ppm in 100 s counting time. Therefore the ARL PERFORM[®]X is well suited for the analysis of typical waste water or industrial water.

Using this mid-power XRF spectrometer allows also the installation to be simpler because no external water is necessary for X-ray tube and anode cooling. Therefore neither tap water, nor a water cooler is required.

Furthermore, operation is made easy through the most advanced state-of-the-art Thermo Scientific OXSAS WDXRF software which operates under the latest Microsoft Windows[®] 10 package.

Table 1: Analysis parameters and typical limits of detection (100 s counting time)

| Element | Line | Crystal | Detector | Collimator | LoD (ppm) |
|---------|------------|---------|----------|------------|-----------|
| As | K β | LiF220 | SC | Fine | 1.1 |
| Ba | L α | LiF200 | FPC | Coarse | 0.9 |
| Cd* | K α | LiF200 | SC | Coarse | 1.1 |
| Co | K α | LiF200 | SC | Coarse | 0.3 |
| Cr | K α | LiF200 | FPC | Coarse | 0.3 |
| Cu | K α | LiF200 | SC | Coarse | 0.2 |
| Hg | L α | LiF200 | SC | Fine | 0.6 |
| Ni | K α | LiF200 | SC | Coarse | 0.2 |
| Pb | L β | LiF200 | SC | Coarse | 0.4 |
| Zn | K α | LiF200 | SC | Coarse | 0.2 |

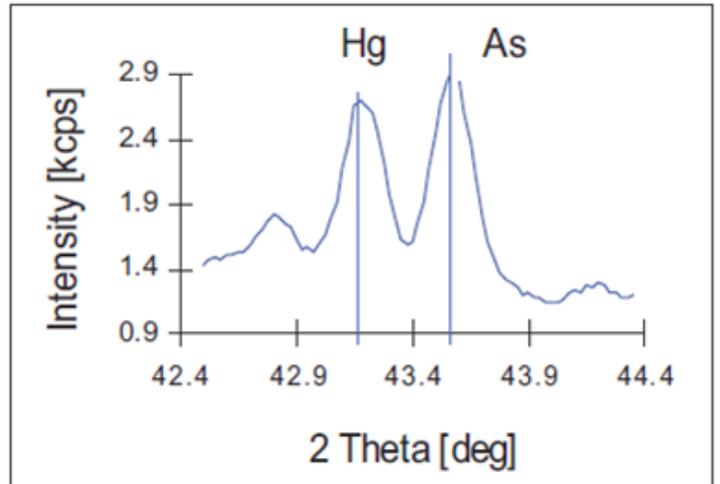
FPC= flow proportional counter

SC = scintillation counter

* = Cd is analyzed with a primary beam filter to suppress overlapping lines due to the rhodium anode of the X-ray tube

For guaranteed values the above limits of detection should be doubled.

Figure 1 : XRF scan showing traces of Hg and As in water



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